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(54) Abstract Title
Water-soluble pouch containing composition

(57) A particulate component of a composition, especially a detergent composition, is contained in a compartment of a pouch formed from a stretchable water-soluble, water-disintegrating or water-dispersable material of non-uniform thickness. A film of the material may be placed in a mould and vacuum formed to make an open compartment, the part of the film in the bottom of the mould, furthest from the points of closing, being stretched more than in the top part. Particulate component to fill at least 95% of the compartment may then be introduced and the compartment closed. The surface area of the compartment may be reduced during or after closure by heat shrinking. The film may be elastic and may comprise polyvinyl alcohol polymer.

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Pouched Compositions

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Technical Field

The present invention relates to compositions in a pouch having a compartment enclosing a particulate component, the compartment being formed from a stretched material of non-uniform thickness.

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Background to the Invention

Cleaning compositions nowadays come in a number of product forms, such as granules, liquids and tablets, each form having its advantages and disadvantages.

15 Recently, tablets have gained renewed interest, mainly because they are easy to handle for the consumer and easy to dose ('unit dose'). To make tablets storage stable and to prevent breakage of the tablets during handling, the ingredients need to be strongly compressed together and generally binding agents are needed to ensure the tablets do not break. This can reduce their solubility and dispersibility which is undesirable for
20 the consumers, both from a performance point of view and from a machine or fabric residue point of view.

Thus, alternative ways and better ways to provide easy to handle, unit dose products which do not dust or break, but dissolve complete and fast, are desirable.

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The inventors have now found an improved method to make a product addressing the above problems, namely by incorporating a particulate product in a (partially) water-soluble or -disintegrating or -dispersable pouch in a specific way, such that the above requirements are fulfilled.

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Pouches for detergents as such are known in the art to be useful to provide unit dose compositions to be delivered to the wash. They typically are described as bag-shape pouches, loosely containing the product. These pouches can be made of water permeable material or water-soluble material. However, the solubility of these
5 pouched products is not always satisfactory.

The inventors have now found improved pouched particulate compositions with improved dissolution, namely pouched compositions having a compartment made from stretchable water-soluble, water-dispersible or water-disintegrating material of
10 non-uniform thickness, this material being stretched around the particulate component (of the composition) in the compartment. The component of the compositions, and preferably the composition as a whole, is thus typically tightly enclosed in the compartment. This way of incorporating the component in a compartment (and of incorporating a composition in a pouch) results in an improved and/ or controlled
15 dissolution in water, e.g. faster and/ or more complete, time-controlled, whilst the pouched composition is storage stable. It is believed that due to the compartment having a non-uniform thickness and being tightly pressed against the component or composition as a whole, the water penetrates through or dissolves quickly the compartment material, in particularly the thinnest part thereof, whilst during storage
20 the remaining of the material of the compartment is still suitable to protect the product against moisture.

Moreover, because the component is tightly enclosed, improved stability upon storage is achieved, because reduced interaction of the ingredients in the compoennt is
25 achieved, similar to compacted tablets. However, because the component and also the composition is in particulate, free-flowing form and not tablets, the dissolution of the components, and composition, into wash water is much better.

Furthermore, improved process are provided to form the pouched composition as
30 described above, such that the component of the composition is enclosed by a stretched, non-uniform compartment material.

Summary of the Invention

The present invention provides a composition in a pouch, the composition comprising a particulate component, the pouch comprising a compartments, whereby the
5 component is present in the compartment and the compartment is formed from a stretchable water-disintegrating, water-soluble or water-dispersable material of non-uniform thickness.

The non-uniform thickness of the compartment material provides very rapid and/ or
10 controlled release of component into water, whilst still ensuring storage stability of the pouched composition or component thereof.

In particular, the composition in a pouch of the invention is obtainable by a process comprising the steps of:

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- a) stretching a stretchable material, preferably a film, forming an open compartment, preferably by introducing the material in a mould and stretching the material into the mould to form the open compartment in the shape of the mould;
 - 20 b) introducing a particulate component in the open component, such that at least 95% of the volume of the open compartment is filled with the component;
 - c) subsequently closing the open compartment.
- 25 Typically, in step b) the open compartment is filled 100% or even overfilled, and when closing the open compartment, the material thereof remains stretched or is further stretched.

The component is typically tightly packed so that the density of the component is
30 increased, i.e. so that the density of the component after closing the compartment is higher than the bulk density of the component prior to incorporation in the

compartment, whilst the still free-flowing, which can be noted when the pouch is removed. This provides the benefits as set out above.

5 The composition in the pouch is preferably such that the bulk density of the composition after closing the compartment is 5% to 45% or even to 35%, or it may be preferred that this is 5% to 30% or even 10% to 25% higher than the average of the bulk density of the components prior to introduction into the compartment.

10 Preferably, the pouch as a whole is water-soluble. Preferably the composition is a cleaning composition.

The invention also provides processes for making the pouched compositions of the invention, including the process described above.

15 Detailed description of the invention

Pouch and compartment thereof

20 The pouch herein comprises a closed structure enclosing a volume space which comprises the composition. Thus, the pouch can be of any form, shape and material which is suitable to hold the composition prior to use, e.g. without allowing the release of the composition from the pouch prior to contact of the pouched composition to water. The exact execution will depend on for example the type and amount of the composition in the pouch, the number of compartments in the pouch, the characteristics required from the pouch to hold, protect and deliver or release the compositions.

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The pouch may be of such a size that it conveniently contains either a unit dose amount of the composition herein, suitable for the required operation. For example, when the composition is a cleaning composition, the amount in the pouch can be such that it is suitable for one wash, or only a partial dose, to allow the consumer greater flexibility to vary the amount used, for example when the pouched composition is a

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detergent composition, it may depend on the size and/ or degree of soiling of the wash load.

5 In one preferred embodiment, the pouched composition has a clearly distinguishable top side and bottom side. Preferably, the pouched composition is formed in a mould with a round or flat bottom and round walls. Thus, preferred is also that the pouched composition is a spheroid or more preferably cylinder-shaped.

10 The pouch has one or more compartments, whereof at least one contains a particulate component. In one execution, more than one particulate component is present and all particulate components are present in one and the same compartment of the pouch. It may be preferred that one or more additional compartments for liquid component are present in addition to the compartment comprising the particulate component (s).

15 However, it may be preferred that there is only one compartment in the pouch, thus containing the composition as a whole, which may consist of the particulate component, or may comprise additional particulate or liquid components. This reduces the material needed to form the compartment and pouch.

20 The compartment of the pouch herein also has a closed structure made of a material as described herein, enclosing a volume space, holding the particulate component. Thus, the compartment is made such that it is suitable to hold the component prior to use, e.g. without allowing the release of the components from the compartment prior to contact of the pouched composition to water. The compartment can have any form
25 or shape, depending on the nature of the material of the compartment, the nature of the component or composition, the intended use, amount of the component etc. If more than one compartment is present, the compartments are linked or connected to one another by any means, for example sealed by heat sealing or by wetting sealing, glued by any known glue material, as also described hereinafter.

The compartment is made of water-dispersable, water-disintegrating or preferably water-soluble material, preferably a film material which is stretchable. Preferably, the pouch as a whole is made of a material which is stretchable. This stretchable material is stretched to form the open compartment shape which is filled for more than 95% by
 5 volume or even 100% or even over filled. Moreover, the material is preferably elastic, to ensure the tight packing and to ensure no (additional) head space can be form after closure of the compartment-

Preferred stretchable materials have a maximum stretching degree of at least 150%,
 10 preferably at least 200%, more preferably of at least 400% as determined by comparison of the original length of a piece of material with the length of this piece of material just prior to rupture due to stretching, when a force of at least 1 Newton is applied. Preferably, the material is such that it has a stretching degree as before, when a force of at least 2Newton, or even at least 3 Newton is used. Preferably, it has this
 15 stretching degree when a force of the above lower limits is used, but not more than 20 Newton, or even 12 Newton, or even 8 Newton.

For example, a piece of film with a length of 10 cm and a width of 1 cm and a thickness of 40 microns is stretched lengthwise with a force of for example
 20 2.8Newton and thus an increasing stress, up to the point that it ruptures. The extent of elongation just before rupture can be determined by continuously measuring the length and the degree of stretching can then be calculated. For example, this piece of film with an original length of 10 cm can be stretched with a force of 2.8 Newton to 52 cm (just before rupture) and thus has a maximum stretching degree of 520% (at a
 25 force of at least 2Newton, namely 2.8 Newton).

The force to stretch such a piece of film (10 cm x 1 cm x 40 microns) to a degree of 200% should preferably be at least 1 Newton, preferably at least 2 Newton, more preferably at least 2.5 or even 3 Newton, and preferably no more than 20 Newton,
 30 preferably less than 12 Newton, most preferably less than 8 Newton. This in particular ensures that the elastic force remaining in the film after forming is high

enough to immobilize the powders within the pouch, but not too high to easily mould and form a pouch from it.

As is clear from the definition herein, the stretchable material is defined by a degree of stretching measured when it is not present in the closed compartment. However, as
 5 said above, the material is stretched when forming the compartment. This can for example be seen by printing a grid onto the material, e.g. film, prior to stretching, then forming a compartment with the component from this material with grid. It can be seen that squares of the grid are elongated and thus stretched.

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The elasticity of the stretchable material of the compartment and preferably the pouch as a whole is herein typically defined as the 'elasticity recovery'. This can be determined by stretching the material (for example to an elongation of 200%, as set out above) and measuring the length of the material after release of the stretching
 15 force. For example a piece of film of a length of 10 cm and width 1 cm and thickness of 40 microns is stretched lengthwise to 20 cm (200% elongation) with a force of 2.8Newtons (as above), and then the force is removed. The film snaps back to a length of 12 cm, which means 80% elastic recovery.

20 Preferably, the compartment material has an elasticity such that the elastic recovery is from 20% to 100%, more preferably from 50% or from 60% or more preferably from 75% or even 80% to 100%.

Thus, the material of the compartment (and preferably the pouch as a whole) is
 25 stretched during formation and/ or closing of the compartment or pouch, such that the resulting pouched composition has a compartment or pouch which is at least partially stretched. The stretching of the material of the compartment when forming the compartment may be done by any means for example by applying a force on the material, including the use of an vacuum, optionally while heating the material.

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Typically and preferably, the degree of stretching is non-uniform over the compartment or pouch, due to the formation and closing process. For example, when a film is positioned in a mould and an open compartment is formed by vacuum forming (and then filled with the components and then closed) the part of the film in the bottom of the mould, furthest removed from the points of closing, will be stretched more than in the top part. The material of the compartment has typically a thickness variation from 10% to 1000%, preferably 20% to 600%, or even 40% to 500% or even 60% to 400%. This can be measured by any method, for example by use of an appropriate micrometer. This can be measured with a pair of calipers such as available from Mitutoyo Uk Ltd, under no. CD-6"CP.

Therefore, it may be preferred that the component to be delivered *first* to the water is comprised in a bottom layer of the compartment, and a component which is to be delivered to the water at a later stage is comprised in a subsequent layer, closer to the top of the compartment. Alternatively, or in addition, it may be preferred that the least moisture sensitive component is comprised in the bottom layer of the compartment and a more moisture sensitive component is comprised in a subsequent or top layer.

Material of pouch and compartment

- Preferably, the composition is a composition to be delivered to water and thus, the pouch and the compartment (s) thereof are designed such that at least one or more of the components is released at, or very shortly after, the time of addition to the water. Thus it is preferred that the compartment and preferably the pouch is formed from a material which is water-dispersible or more preferably water-soluble.
- In one preferred embodiment, the particulate component is delivered to the water within 3 minute, preferably even within 2 minutes or even within 1 minute after contacting the pouched composition to water.

Preferred water-dispersable material herein has a dispersability of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out hereinafter using a glass-filter with a maximum pore size of 50 microns.

More preferably the material is water-soluble and has a solubility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out hereinafter using a glass-filter with a maximum pore size of 20 microns, namely:

- 5 Gravimetric method for determining water-solubility or water-dispersability of the material of the compartment and/or pouch:
50 grams \pm 0.1 gram of material is added in a 400 ml beaker, whereof the weight has been determined, and 245ml \pm 1ml of distilled water is added. This is stirred vigorously on magnetic stirrer set at 600 rpm, for 30 minutes. Then, the mixture is
10 filtered through a folded qualitative sintered-glass filter with the pore sizes as defined above (max. 20 or 50 micron). The water is dried off from the collected filtrate by any conventional method, and the weight of the remaining polymer is determined (which is the dissolved or dispersed fraction). Then, the % solubility or dispersability can be calculated.
15 Preferred materials are polymeric materials, preferably polymers which are formed into a film or sheet. The material in the form of a film can for example be obtained by casting, blow-molding, extrusion or blow extrusion of the polymer material, as known in the art.
20 Preferred polymer copolymers or derivatives thereof are selected from polyvinyl alcohols, polyvinyl pyrrolidone, polyalkylene oxides, (modified) cellulose, (modified)cellulose-ethers or -esters or -amides, polycarboxylic acids and salts including polyacrylates, copolymers of maleic/acrylic acids, polyaminoacids or
25 peptides, polyamides including polyacrylamide, polysaccharides including starch and gelatine, natural gums such as xanthum and carragum. Preferably, the polymer is selected from polyacrylates and acrylate copolymers, including polymethacrylates, methylcellulose, sodium carboxymethylcellulose, dextrin, maltodextrin, ethylcellulose, hydroxyethyl cellulose, hydroxypropyl methylcellulose,; most preferably polyvinyl
30 alcohols, polyvinyl alcohol copolymers and/ or hydroxypropyl methyl cellulose(HPMC).

The polymer may have any weight average molecular weight, preferably from about 1000 to 1,000,000, or even from 10,000 to 300,000 or even from 15,000 to 200,000 or even from 20,000 to 150,000.

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Mixtures of polymers can also be used. This may in particular be beneficial to control the mechanical and/or dissolution properties of the compartment or pouch, depending on the application thereof and the required needs. For example, it may be preferred that a mixture of polymers is present in the material of the compartment, whereby one
10 polymer material has a higher water-solubility than another polymer material, and/ or one polymer material has a higher mechanical strength than another polymer material. It may be preferred that a mixture of polymers is used, having different weight average molecular weights, for example a mixture of PVA (or a copolymer thereof) and/ or HPMC of a weight average molecular weight of 10,000- 40,000, preferably
15 around 20,000, and of PVA (or copolymer thereof) and/ or HPMC with a weight average molecular weight of about 100,000 to 300,000, preferably around 150,000.

Also useful are polymer blend compositions, for example comprising hydrolytically degradable and water-soluble polymer blend such as polylactide and polyvinyl alcohol,
20 achieved by the mixing of polylactide and polyvinyl alcohol, typically comprising 1-35% by weight polylactide and approximately 65-99 by weight polyvinyl alcohol, if the material is to be water-dispersable, or water-soluble.

It may be preferred that the polymer present in the material of the compartment is
25 from 60-98% hydrolysed, preferably 80% to 90%, to improve the dissolution of the material.

Most preferred are materials which are water-soluble stretchable and elastic material comprising PVA polymer having properties such as the PVA films sold under the
30 trade reference M8630, as sold by Chris-Craft Industrial Products of Gary, Indiana, US.

Preferably, the level of a type polymer (e.g. commercial mixture) in the film material, for example PVA polymer, is at least 60% by weight of the material or film, preferably at least 60% or even at least 70% or even at least 80 or 90%. The upper
5 level is up to 100%, but typically 99% or even 98% by weight.

The material herein may comprise other additive ingredients then the polymer or polymer material. For example, it may be beneficial to add plasticisers, for example glycerol, ethylene glycol, diethyleneglycol, propylene glycol, sorbitol and mixtures
10 thereof, additional water, disintegrating aids. It may be useful when the pouched composition is a detergent composition, that the pouch or compartment material itself comprises a detergent additive to be delivered to the wash water, for example organic polymeric soil release agents, dispersants, dye transfer inhibitors.

15 The material in the form of a film may be coated, preferably only one-sided, with any coating method and with any coating agent, depending on the required properties; for example, it may be beneficial to coat the film such that the compartment or pouch or composition therein, is more storage stable and/or less sensitive to moisture and/ or acts as a improved moisture barrier.

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A very useful form is to coat the material or film on one side with a coating that slows the dissolution of the film, prior to forming of the compartment and thus prior to stretching the material or film. Then, by stretching the material or film, the coating is stretched as well, resulting in cracks in the coating and/ or uneven distribution of the
25 coating over the material and thus over the compartment. This then ensures still stability against moisture during storage, whilst the presence of cracks or the uneven distribution still ensures the required dissolution in use. Hence it is possible to make a pouched composition that is resistant to being handled with wet fingers when it is picked up at the sides but will still release product rapidly when it is immersed in
30 water due to film rupture at the thinnest points.

Any coating material can be used, particular useful are hydrophobic coatings, or polymers with a low water-solubility, lower then defined herein before.

5 The compartment material may be shrinkable material, so that the surface area can be reduced during or subsequent to closing the open compartment by shrinking the material.

10 Preferably, the open compartment is closed with a piece of the same material as the material of the open compartment. The closing material, and thus preferably also the open compartment material or shape material, is preferably thermoplastic so that it can be closed by heat-sealing. Alternatively, a thermoplastic coating may be provided, either over the whole material or just in the areas where seals are to be formed. The sealing can also be made by solvent welding or wetting sealing. Suitable heat-sealable materials include polyvinyl alcohol, polyvinyl acetate, polyvinyl pyrrolidone, 15 polyethylene oxide, acrylic resins and mixtures thereof, in particular polyvinyl alcohols.

20 Compositions and components thereof

The composition of the invention is present in a pouch and is herein also referred to as pouched composition. The composition comprises at least one particulate component. Generally, the particulate component is a powder, granular, extrudate or flake component. Preferred are cleaning compositions, such as laundry detergents, 25 dish washing detergent, and fabric care compositions such as conditioners and other rinse additives.

The pouched composition herein is obtainable by a method whereby the component is introduced in the open compartment (preferred processes herefor being described 30 below) such that the open compartment is almost completely filled, typically such that at least 95% of the volume of that open compartment (shape), preferably at least 98%

or even at least 100% of the open compartment shape is filled, prior to closing said open compartment shape. More preferably, the open compartment (shape) is overfilled with the components, i.e. that the volume of the components is more than 100% of the volume of the shape, preferably more than 105% or more preferably
5 more than 110% or even more than 115%.

The composition in the pouch herein can for example be obtainable by a process involving:

- 10 a) stretching a stretchable material, preferably a film, thereby forming an open compartment, preferably by introducing the material in a mould and stretching the material into the mould to form the compartment in the shape of the mould;
- b) introducing a particulate component in the open component, such that
15 at least 95% but preferably at least 100% of the volume of the open compartment is filled with the component;
- c) subsequently closing the open compartment.

In one preferred process, step c), or b) and c) of the process above may be performed
20 under reduced pressure, lower than atmospheric pressure, preferably by applying a vacuum, so that after filling and closing under reduced pressure or vacuum, a tightly packed compartment is obtained.

The compartment can be closed by further stretching the material and closing the
25 compartment, but preferably the open compartment is closed with an additional piece of material, which may be the same type of material. Preferably, the open compartment and the closing material are both films of stretchable material, preferably both water-soluble materials. They can then be sealed together by any means, to ensure closure of the compartment, for example by wet welding or more preferably
30 heat sealing.

Typically, the incorporation of the component into the compartment is such that the bulk density of component after closing the compartment is increased with 5% to 45% preferably to 35%, preferably 5% or even 10% to 30% or even to 25%, or even 8% to 20% or even to 15%, compared to the bulk density of the component prior to incorporation in the compartment.

The bulk density of a component prior to incorporation in the pouched composition can be determined by the Repour Cup method, as described in ISO 3424- 1975-E.

10 The bulk density of the component after closing of the compartment can be determined by a method of volume displacement. For example, a vessel with a wide neck and an off-take arm is filled with a solvent of known density, which must not affect the compartment material, up to the level of the collecting arm. The component in the compartment to be tested is accurately weighed and then immersed in the liquid, 15 for example by using a piece of thin metal wire. The amount of liquid that is displaced is the liquid leaving the vessel through the arm and this is collected and carefully weighed. The replaced volume of displaced liquid is easily calculated from this weight and the known density of the liquid. Then, the volume replacement due to the material of the compartment (rather than the component therein) can be measured or 20 calculated. This is deducted from the volume replacement as measured in the above test, to obtain the volume replacement of the actual component. The density of the component as it is in the closed compartment can then be calculated. (Errors associated with the thin wire used to immerse the pouch are minor and are not taken into account.) Depending on the material of compartment, a suitable liquid can be 25 selected. For example, for water-soluble material such as PVA, preferred liquid is glycerol. This is because the compartment may contain microscopic pinholes in the film as a result of the stretching. Using a viscous solvent such as glycerol will minimise any errors due to liquid seeping into the pouch. Also useful are nonionic surfactants such as Neodol 23-5.

The tight packing can be done by increasing the packing efficiency and reducing space between the particles of the component, for example by vibration of the components in the open compartment, allowing the component particles to settle for a period of time, modestly increasing pressure provided that the component and typically the composition as a whole remains free-flowing, for example by applying a pressure of up to 20Mpa, preferably up to 10Mpa or more preferably up to 5Mpa or even up to 2MPa, if any pressure is used.

It may be preferred that the composition in the pouch comprises different components, for example more than one particulate component, or also a liquid component. Then, it may be preferred that the different components are comprised in different compartments, and it may be preferred that the thickness of the material of one compartment is different to another compartment, so that controlled or sequential release of the component therein can be achieved.

When used herein, 'different' component means that one component has at least one different chemical property, for example at least one different ingredient, than the other component or components, or one component has at least one different physical property than another component or component. Examples are described herein after.

The composition and the component herein can be any particulate composition, in particular any free-flowable granular or powder composition to be delivered to, and active in water.

Preferred compositions are beverages, edible compositions, pharmaceutical compositions, personal care compositions, cleaning compositions, fabric care or conditioning compositions; most preferably, the compositions herein are cleaning compositions or fabric care compositions, preferably hard surface cleaners, more preferably laundry or dish washing compositions, including detergents, pretreatment or soaking compositions or fabric conditioners, and other rinse additives.

When used in cleaning compositions the component can contain any active cleaning ingredient. In particular preferred are active ingredients such as surfactants, chelating agents, builders, enzymes, perfumes, bleaches, bleach activators, fabric softeners,
5 fabric conditioners, antibacterial agents, effervescence sources, brighteners, photo-bleaches.

Fabric care compositions or rinse additives preferably comprise at least one or more softening agents, such as quaternary ammonium compounds and/ or softening clays,
10 and preferably additional agent such as anti-wrinkling aids, perfumes, chelants, fabric integrity polymers.

Generally, water is present at a level of 0% to 10%, more preferably from 0.2% to 5% or even 0.2% to 3% or even from 0.5% to 2% by weight of the compartment,
15 preferably of the pouch.

Although the nature of the pouched composition is such that it readily dissolves or disperses into the water, it may be preferred that disintegrating agents such as effervescence sources, water-swellaable polymers or clays are present in the pouch or
20 compartment material it self, and/ or in the composition therein, in particular effervescence sources based on an acid and a carbonate source. Suitable acids include the organic carboxylic acids such as fumaric acid, maleic acid, malic acid, citric acid; suitable carbonate sources include sodium salts of carbonate, bicarbonate, percarbonate. Preferred levels for the disintegrating aids or effervescence sources or
25 both are from 0.05% to 15% or even from 0.2% to 10% or even from 0.3 to 5% by weight pouched composition.

The component comprises at least an amount of at least one particulate compound, but typically the component comprises at least two particulate compounds, preferably
30 thoroughly mixed to form the component. Because the component in the compartment of the pouch is tightly packed, the interaction between the different

ingredients is reduced. This allows the incorporation in a component of even incompatible ingredients, if required. Thus, the component may comprise one or more enzymes and a bleach such as a peroxygen bleach, such as a salt of percarbonate. It may however be beneficial to include the bleaching agents, but in particular the peroxygen bleach, in a different component than the enzymes and/ or other hygroscopic materials or anhydrous or hydratable materials including overdried materials such as aluminosilicates, anhydrous salts or acids.

When the pouched compositions is such that it has a clear top and bottom side and different component are present in the form of layers, it can also be beneficial to include in the bottom layer non-gelling detergent ingredients, such as water-soluble salts and acids, including for example effervescing salts and acids such as carbonate salts and organic carboxylic acids such as citric acid, and in a higher layer or the top layer potential gelling ingredients such as anionic and nonionic surfactants. At the bottom part, the compartment material is typically more stretched, which will lead to the bottom region to be dissolved prior to the top region of the pouched composition.

It may even be possible that part or all of the ingredients in the component are not pre-granulated, such as agglomerated, spray-dried, extruded, prior to incorporation into the compartment, and that the component is a mixture of dry-mixed powder ingredients or even raw materials. Preferred may be that for example less than 60% or even less than 40% or even less than 20% of the components are free-flowable pre-granulated granules

Also, it has been found that it is beneficial for the cleaning performance, when the cleaning compositions herein or the material of the compartment or pouch, preferably both the composition and said material, comprise one or more chelating agents, in particular phosphonate- and/or carboxylate-containing chelating agents, in particular EDDS or EDTA or HEDP.

It has also been found that the presence in the wash water of high levels of certain dissolved compartment or pouch material having free hydroxy groups can have a negative effect on the removal of clay stains, under certain wash conditions.

Therefore, it is not only beneficial to use as little compartment material as possible
 5 and thus to use preferably a pouch with only one compartment, but it has also been found that it is beneficial to incorporate in the composition or the pouch material a polyalkoxylated compound, preferably a polyalkoxylated alcohol, preferably having an average alcohol carbon chain length of 11 to 24, preferably 12 to 20 or even 14 to 18, and an average alkoxylation degree of at least 20 or even at least 40 or even at least
 10 70. Highly preferred are from 0.1% to 8%, or even from 0.5 to 5% or even from 0.8% to 3% by weight of the pouched composition of such a compound; highly preferred is TAE80.

Another preferred ingredient is a perhydrate bleach, such as salts of percarbonates,
 15 particularly the sodium salts. It has been found that in when the pouch or compartment comprises a material with free hydroxy groups, such as PVA, the preferred bleaching agent comprises a percarbonate salt and is preferably free from any perborate salts, and also borate salts. The borates and perborates interact with these hydroxy-containing materials this can reduce the dissolution of the materials and
 20 may also result in reduced performance.

Preferred are also organic peroxyacid bleach precursor or activator compound, such as alkyl percarboxylic precursor compounds of the imide type include the N-,N,N¹N¹ tetra acetylated alkylene diamines wherein the alkylene group contains from 1 to 6
 25 carbon atoms, particularly those compounds in which the alkylene group contains 1, 2 and 6 carbon atoms such as tetraacetyl ethylene diamine (TAED), sodium 3,5,5-trimethyl hexanoyloxybenzene sulfonate (iso-NOBS), sodium nonanoyloxybenzene sulfonate (NOBS), sodium acetoxymethyl benzene sulfonate (ABS) and pentaacetyl glucose, but also amide substituted alkyl peroxyacid precursor compounds.

Highly preferred ingredient for use herein are one or more enzymes. Preferred enzymes include the commercially available lipases, cutinases, amylases, neutral and alkaline proteases, cellulases, endolases, esterases, pectinases, lactases and peroxidases conventionally incorporated into detergent compositions. Suitable

5 enzymes are discussed in US Patents 3,519,570 and 3,533,139. Preferred commercially available protease enzymes include those sold under the tradenames Alcalase, Savinase, Primase, Durazym, and Esperase by Novo Industries A/S (Denmark), those sold under the tradename Maxatase, Maxacal and Maxapem by Gist-Brocades, those sold by Genencor International, and those sold under the

10 tradename Opticlean and Optimase by Solvay Enzymes. Preferred amylases include, for example, α -amylases obtained from a special strain of *B licheniformis*, described in more detail in GB-1,269,839 (Novo). Preferred commercially available amylases include for example, those sold under the tradename Rapidase by Gist-Brocades, and those sold under the tradename Termamyl, Duramyl and BAN by Novo Industries

15 A/S. Highly preferred amylase enzymes maybe those described in PCT/ US 9703635, and in WO95/26397 and WO96/23873. The lipase may be fungal or bacterial in origin being obtained, for example, from a lipase producing strain of Humicola sp., Thermomyces sp. or Pseudomonas sp. including Pseudomonas pseudoalcaligenes or Pseudomas fluorescens. Lipase from chemically or genetically

20 modified mutants of these strains are also useful herein. A preferred lipase is derived from Pseudomonas pseudoalcaligenes, which is described in Granted European Patent, EP-B-0218272.

Another preferred lipase herein is obtained by cloning the gene from Humicola lanuginosa and expressing the gene in Aspergillus oryza, as host, as described in

25 European Patent Application, EP-A-0258 068, which is commercially available from Novo Industri A/S, Bagsvaerd, Denmark, under the trade name Lipolase. This lipase is also described in U.S. Patent 4,810,414, Høge-Jensen et al, issued March 7, 1989.

Preferred are also anionic surfactants, which include salts (including, for example,

30 sodium, potassium, ammonium, and substituted ammonium salts such as mono-, di- and triethanolamine salts) of the anionic sulfate, sulfonate, carboxylate and sarcosinate

- surfactant, preferably linear or branched alkyl benzene sulfonate, alkyl sulphates and alkyl ethoxysulfates, isethionates, N-acyl taurates, fatty acid amides of methyl tauride, alkyl succinates and sulfosuccinates, monoesters of sulfosuccinate (especially saturated and unsaturated C₁₂-C₁₈ monoesters) diesters of sulfosuccinate (especially
- 5 saturated and unsaturated C₆-C₁₄ diesters), N-acyl sarcosinates. Highly preferred is that when anionic surfactants are present, at least one alkyl sulphate surfactant is present, preferably a branched alkyl sulphate surfactant, preferably at a level of 1% to 20% or even to 15% by weight of the component or composition.
- 10 Also preferred are nonionic surfactants such as nonionic surfactant selected from the classes of the nonionic condensates of alkyl phenols, nonionic ethoxylated alcohols, nonionic ethoxylated/propoxylated fatty alcohols, nonionic ethoxylate/propoxylate condensates with propylene glycol, and the nonionic ethoxylate condensation products with propylene oxide/ethylene diamine adducts.
- 15 Cationic surfactants and softening agents may also be included herein, for example quaternary ammonium surfactants and softening agents, and choline ester surfactants.
- Colouring agent such as iron oxides and hydroxydes, azo-dyes, natural dyes, may also
- 20 be present in the composition or preferably in the compartment or pouch material, preferably present at levels of 0.001% and 10% or even 0.01 to 5% or even 0.05 to 1% by weight of the pouched composition.
- 25 Example 1:
- A piece of Chris-Craft M-8630 film, 38 microns thick, is placed on top of a mould and fixed in place. The mould consists of a cylindrical shape with a diameter of 45 mm and a depth of 25 mm. A 1 mm thick layer of rubber remains present around the edges of the mould. The mould has some holes in the mold material to allow a
- 30 vacuum to be applied.

A vacuum is applied to pull the film into the mold and pull the film flush with the inner surface of the mould. 40 g of a detergent powder mix comprising percarbonate salt and water-soluble salts and organic acids, typically carbonate salts, citric acid and/ or
 5 citrate, enzymes, bleach activator and surfactants is poured into the mould. This powder mix has a bulk density of 860 g/l prior to being poured into the mould. This is slightly vibrated. The mould is filled between 105% to 115%.

Next, a sheet of the same M-8630 film is placed over the top of the mould with the
 10 powder and sealed to the first layer of film by applying an annular piece of flat metal of an inner diameter of 46 mm and heating that metal under moderate pressure onto the ring of rubber at the edge of the mould, to heat-seal the two pieces of film together. The metal ring is typically heated to a temperature of 140 - 146 °C and applied for up to 5 seconds. The film is stretched during this process, which can be
 15 visualised by using in this example a film material with a grid on it. The thickness variation of the film is between 20 and 40 microns, the bottom being 20 microns, the top being 40 microns and the sides varying between 20 and 40 microns.

The bulk density of the pouched composition was then tested by the method described
 20 above and found to be 1020 g/l.

Pouches made by the above method released product on immersion in 5 liters of 10 °C water in less than 10 seconds.

25 A 10 cm x 1 cm x 40 microns piece of this film material itself was tested for elasticity and stretchability, using a 2.8Newton force, as described above. The maximum stretching degree was 520 and the elastic recovery was 85%.

Example 2

30 The example 1 is repeated, filling the open pouch in the mould first with 17.5 g of detergent component comprising 8 gram sodium percarbonate and 4 gram citric acid

and 5.5 gram sodium carbonate and then 17.5 gram of a component comprising 0.1 gram enzymes, 5 gram anionic surfactant and 10 gram zeolite, 2.4 gram of perfume, bleach activator (TAED), brightners, and other minor additives.

5 **Example 3**

- The example 1 is repeated, filling the open pouch in the mould first with 30 g of detergent component comprising sodium percarbonate and citric acid and sodium carbonate enzymes anionic surfactant and zeolite, perfume, bleach activator (TAED), brightners, and other minor additives as in example 2, the balance being sodium sulphate filler salts, and then a second layer of 5 gram of a fabric softening clay was added.
- 10

The following are detergent compositions which may also be suitable incorporated in a pouch of the invention:

	A	B	C	D	E	F	G	H	I
<u>Spray-dried Granules</u>									
LAS	10.0	10.0	15.0	5.0	5.0	10.0	-	-	-
TAS	-	1.0	-				-	-	-
branched or linear synthetic alkyl sulphate surfactant	-	-	2.0	5.0	5.0		-	-	-
cationic surfactant			1.0	1.0			-	-	-
DTPA, HEDP and/or EDDS	0.3	0.3	0.5	0.3			-	-	-
MgSO ₄	0.5	0.5	0.1	-			-	-	-
Sodium citrate	-	-	-	3.0	5.0		-	-	-
Sodium carbonate	10.0	7.0	15.0			10.0	-	-	-
Sodium sulphate	5.0	5.0	-	-	5.0	3.0	-	-	-
Sodium silicate 1.6R	-	-	-	-	2.0		-	-	-
Zeolite A	16.0	18.0	20.0	20.0	-	-	-	-	-

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Claims

1. A composition in a pouch, the composition comprising a particulate component and the pouch comprising a compartment, whereby the component is present in the compartment and the compartment is formed from a stretchable water-disintegrating, water-soluble or water-dispersable material of non-uniform thickness.
5
2. A composition in a pouch according to claim 1 obtainable by a process comprising the steps of:
10
 - a) stretching a stretchable material, preferably a film, forming an open compartment, preferably by introducing the material in a mould and stretching the material into the mould to form the open compartment in the shape of the mould;
15
 - b) introducing a particulate component in the open component, such that at least 95% of the volume of the open compartment is filled with the component;
 - c) subsequently closing the open compartment.
20
3. A composition according to claim 2 whereby the process comprises the step of increasing the bulk density of the component with 5% to 45%, preferably 5% to 35% or even 20%, or even 10% to 25%.
25
4. A composition according to claim 2 or 3 whereby step c) or b) and c) are performed under reduced pressure which is lower than atmospheric pressure, preferably by applying a vacuum.
30
5. A composition according to claim 2 or 3 whereby in step b) the component is introduced in the open compartment such that at least 100%, preferably at

least 110%, of the volume of the open compartment is occupied by the component.

6. A composition in a pouch according to any preceding claim whereby the compartment is formed from an elastic material, preferably a film.
7. A composition according to any of claims 2 to 5 whereby the compartment is formed from a stretchable, shrinkable material and in step c) the surface area of the compartment is reduced during or subsequent to closing the open compartment by shrinking the open compartment and/or compartment, preferably by heat-shrinking.
8. A composition according to any preceding claim whereby the compartment is formed from a material of non-uniform thickness having a variation in thickness of at least 40%, preferably up to 500%.
9. A composition in a pouch according to any preceding claim whereby the stretchable material has a maximum stretching degree of at least 200% and preferably an elastic recovery of from 50% to 100%.
10. A composition in a pouch according to any preceding claim whereby the compartment and the pouch are water-soluble.
11. A composition in a pouch according to any preceding claim whereby the compartment is formed from a film comprising polyvinyl alcohol polymer.
12. A composition in a pouch according to any preceding claim which is a cleaning composition in a pouch, preferably a laundry or dish washing detergent in a pouch.

- 13 A process as in claim 2 for making the pouched product as in any of claims 1 to 12



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Application No: GB 0010220.2
Claims searched: 1-12

Examiner: Stephen Smith
Date of search: 24 November 2000

Patents Act 1977 Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK CI (Ed.R): B8C(CWA1, CWA2, CWS8)

Int CI (Ed.7): B65D 65/46

Other: ONLINE:EPODOC, JAPIO, WPI

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
A	WO 97/00282 A1 (ZENECA)	

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.